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REMARKS

I. STATUS OF THE CLAIMS

Claims 21-52 are pending in this application. No amendments have been made.

II. REJECTION OF CLAIMS 21-52 UNDER 35 U.S.C. § 112, FIRST PARAGRAPH

The Examiner maintains her rejection of claims 21-52 under 35 U.S.C. § 112, first paragraph. The Examiner asserts that the present application “fails to describe specific examples of adhesive polymers that satisfy the criteria of claims 21-26.” Office Action at 3. Specifically, the Examiner takes issue with the description in the specification, in which, she alleges “[t]here is no structure given to this polymer except for the trademark name.” *Id.* The Examiner contends that the functional language in the claims is not sufficient (*i.e.*, without further description of the polymers) and that “one of ordinary skill in the art could not recognize or understand the polymers that satisfy the requirement from the mere recitation of the function.” *Id.*

Applicants respectfully disagree with the Examiner, and traverse the rejection with respect to all claims for at least the following reasons.

First, Applicants respectfully remind the Examiner that she “has the initial burden of presenting by a **preponderance of evidence** why a person skilled in the art would not recognize in an applicant’s disclosure a description of the invention defined by the claims.” M.P.E.P. § 2163 (emphasis added) (citing *In re Wertheim*, 541 F.2d 257, 262 (CCPA 1977)). In the present case, the Examiner has not met her burden of proof and has not shown that one of ordinary skill in the art would not recognize Applicants’ possession of the claimed invention based on the description of the polymers’ properties.

Furthermore, M.P.E.P. 2163 dictates “Guidelines” for assisting Examiners in the evaluation of any patent application for compliance with the written description requirement of 35 U.S.C. § 112, first paragraph. M.P.E.P. 2163 explicitly states:

The Guidelines do not constitute substantive rulemaking and thus **do not have the force and effect of law**. They are designed to assist Office personnel in analyzing claimed subject matter for compliance with substantive law. **Rejections will be based upon the substantive law** M.P.E.P. 2163 (emphasis added).

Neither in the first Office Action, nor in the present final Office Action, has the Examiner provided any substantive legal support for her written description rejections.

Second, as previously argued, 35 U.S.C. § 112, first paragraph, does not require that Applicants disclose a particular structure for polymers that exhibit the properties recited in the claims. To satisfy the written description requirement, a specification must describe the claimed invention in sufficient detail that one skilled in the art can reasonably conclude that the inventor had “possession” of the claimed invention. See, e.g., *Vas-Cath, Inc. v. Mahurkar*, 935 F.2d 1555, 1563 (Fed. Cir. 1991). Under the law, “possession” may be shown in a number of ways, including by describing an actual reduction to practice, or by describing distinguishing identifying characteristics sufficient to show that the applicant was in possession of the claimed invention. See, e.g., *Pfaff v. Wells Elecs., Inc.*, 525 U.S. 55, 68 (1998).

Thus, the detailed language of the claims themselves is sufficient to demonstrate to one skilled in the art that Applicants had possession of the invention when the application was filed. The present claims recite a cosmetic hair composition that has very specific properties that are understood by one skilled in the art. This is not mere “functional” language, as asserted by the Examiner, but rather, a listing of particular

physical properties of the composition. The recitation of such detail in both the specification and claims leaves no doubt in the mind of one skilled in the art that Applicants knew what they had invented and were in possession of it at the time of filing. Such detail is more than sufficient to meet the written description requirements of 35 U.S.C. § 112, first paragraph. Therefore, the lack of a general polymeric structure in either the specification or claims is not sufficient to render the claims invalid under 35 U.S.C. § 112, first paragraph.

Moreover, possession of the invention within the meaning of 35 U.S.C. § 112, first paragraph, may be demonstrated by the description of an actual reduction to practice. M.P.E.P. § 2163(I). In this case, Applicants have described an actual reduction to practice of the invention in Examples 1 and 4, found on pages 11 and 13, respectively, of the originally-filed specification. The compositions described in these examples exhibit the properties recited in the instant claims.

Finally, the Examiner continues to overlook the fact that the specification **does** provide structural description, despite the fact that a structural description is not required. The specification provides that an example of an adhesive polymer that can be used is “the **branched sulfonic** polymer AQ 1350 sold by the company Eastman.” Specification at 4 (emphasis added). Applicants respectfully submit that the description “branched sulfonic polymer” indicates structure. In addition, Applicants submit herewith U.S. Patent No. 5,543,488 (the ‘488 patent), assigned on its face to Eastman Chemical Company. See Appendix A. Applicants respectfully point out that the ‘488 patent, in particular its Examples 2-4, describes the branched sulfonic polymer AQ 1350. Applicants further submit a copy of “Eastman AQ Branched Polyesters,” a product

information brochure. See Appendix B. Applicants respectfully assert that one skilled in the art would have access to and understand the '488 patent, just as one skilled in the art would have access to the full description of AQ 1350, as with other Eastman AQ polymers, through the publications of Eastman itself.

Thus, for at least the foregoing reasons, Applicants respectfully submit that the rejection of all claims under 35 U.S.C. § 112, first paragraph is in error and should be withdrawn.

III. REJECTION OF CLAIM 37 UNDER 35 U.S.C. § 112, FIRST PARAGRAPH

The Examiner rejects claim 37 under 35 U.S.C. § 112, first paragraph, for allegedly containing new subject matter, "which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention." Office Action at 4. Specifically, the Examiner contends that there is no support at page 10, 2nd paragraph of the specification for the recitation of "ketones, alkyl acetates, and di-alkoxy substituted alkanes," in claim 37. The Examiner concludes that "[d]escription for two specific compounds in each category [i.e., ketones, alkyl acetates, and di-alkoxy substituted alkanes] is not a support for the entire genus." Office Action at 5. Further, the Examiner rejects Applicants' previous argument that the recitation of at least two species can, in fact, be representative of a genus.

Applicants continue to respectfully disagree with the Examiner and traverse for at least the following reasons.

First, Applicants continue to respectfully assert that the written description requirement for claims to a genus may be met by the disclosure of a representative

number of species within that genus. M.P.E.P. § 2163.05(I). What constitutes a “representative number of species” within a particular genus is dependent on the types of compounds being claimed and their disclosed use. *Id.* In some cases, recitation of a single species has been held to be an adequate description of an entire genus. See *In re Rasmussen*, 650 F.2d 1212, 211 UPSQ 323 (CCPA 1981); and *In re Herschler*, 591 F.2d 693, 200 USPQ 711 (CCPA 1979). Furthermore, Applicants respectfully submit that the Examiner is not recognizing that the specification paragraph in question uses language that is clearly permissive to describe the organic solvents that may be used.

Second, proper written description support does not require the language of the claim to be set forth “*in haec verba*” in the specification. *In re Wright*, 866 F.2d 422, 425 (Fed. Cir. 1989). The Examiner is improperly looking to the scope of the phrase of the specification, rather than considering whether the disclosure of the specification supports claim 37. As Applicants have previously argued, with respect to organic solvents, the recitation of at least two species contained within each genus, combined with the permissive language of the description, provides plenty of support for one of ordinary skill in the art to recognize that the inventors had possession of the claimed subject matter.

Therefore, for at least the foregoing reasons, Applicants respectfully submit that the rejection of claim 37 under 35 U.S.C. § 112, first paragraph is improper and should be withdrawn.

IV. REJECTION OF CLAIMS 21-38 AND 42 UNDER 35 U.S.C. § 102(B)

The Examiner rejects claims 21-38 and 42 under 35 U.S.C. § 102(b) as anticipated in view of U.S. Pat. No. 5,053, 221 to Robertson et al ("Robertson"). In particular, the Examiner states:

See col.2, lines 30-45 for the beads which read on the claimed particles. There is overlap of concentration and particle size. See the paragraph bridging col.s 3-4 for the adhesive polymer and see col.4, lines 18 et seq and col.5, lines 1-60 for the AQ polymers. The polymer disclosed in the patent reads on the claimed adhesive polymer. See the examples for ethanol which read on the solvent, see col5, lines 60 et seq and col.6 for the additives.

Office Action at 6.

Applicants respectfully disagree with the Examiner traverse the rejection for at least the following reasons.

A claim is anticipated under 35 U.S.C. § 102(b) only if each and every element as set forth in the claim is found in a single reference. See *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631 (Fed. Cir. 1987) and M.P.E.P. § 2131.

Furthermore, the identical invention must be set forth in as complete detail as it appears in the claim. See *Richardson v. Suzuki Motor Co.*, 868 F.2d 1226, 1236 (Fed. Cir. 1989) and M.P.E.P. § 2131. Robertson cannot be said to anticipate the present invention because it does not disclose each and every element of the present claims.

First, Robertson does not disclose the presently claimed particles of the invention. Robertson discloses and requires "an effective amount of microsphere particles selected from . . . silica microspheres (beads), polymethylmethacrylate microspheres (beads), and combinations thereof." Robertson at col. 1, lines 54-57.

Thus, Robertson only discloses **spherical** particles, whereas the solid particles of the

presently claimed invention are flakes, platelets, leaflets, fibrils and powders, *i.e.*, **flat** particles. See, *e.g.*, claim 27.

Moreover, Robertson does not anticipate the instant claims because there is no teaching or disclosure that the disclosed compositions in Robertson would exhibit the claimed detachment profile. The Examiner attempts to make an inherency argument by stating that “[t]he office is not equipped to manufacture the composition and then drying the composition so that it satisfies the functional criteria of claims 21-26. Therefore the claims are anticipated, absence of the contrary.” Office Action at 6. However, such a statement by the Examiner does not qualify as a proper inherency argument under M.P.E.P. § 2112. (“Examiner must provide rationale or evidence tending to show inherency”). “Inherency . . . may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient.” *In re Roberson*, 169 F.3d 743, 745 (Fed. Cir. 1999) (citations omitted).

The Examiner’s rejection of the present claims as anticipated is improper in light of the facts at hand. There is no evidence that the AQ polymers disclosed in Robertson -- AQ29, AQ38, and AQ55 -- would provide the instantly claimed detachment profiles, especially since these AQ polymers have T_g values (29°C, 38°C, and 55°C, respectively) much larger than those of AQ 1350 (T_g = 0°C). Additionally, AQ29, AQ38, and AQ55 all have hydroxyl numbers of less than 10, whereas AQ 1350 has a hydroxyl number of 28. To demonstrate the differences between the polymers of Robertson, and those of the presently claimed invention, Applicants also submit the following two product brochures from Eastman Chemicals: “Eastman AQ Polymers” and “Eastman AQ 55S and Eastman AQ 38S.” See Appendix C and D.

It is clear that the presently claimed polymers would not include those disclosed in Robertson, and thus there is no support for the Examiner's anticipation rejection. There is simply no evidence that the AQ polymers cited in Robertson do or would lead to compositions with the presently claimed detachment profile.

Therefore, for at least the foregoing reasons, Applicants respectfully submit that the rejection of claims 21-38 and 42 under 35 U.S.C. § 102(b) is improper and should be withdrawn.

V. CONCLUSION

In view of the foregoing amendments and remarks, Applicant respectfully requests the reconsideration and reexamination of this application and the timely allowance of the pending claims.

Please grant any extensions of time required to enter this response and charge any additional required fees to our deposit account 06-0916.

Respectfully submitted,

FINNEGAN, HENDERSON, FARABOW,
GARRETT & DUNNER, L.L.P.

Dated: June 9, 2004

By: Thalia V. Warnement
Thalia V. Warnement
Reg. No. 39,064

ENCLOSURES:

Appendix A: U.S. Patent No. 5,543,488
Appendix B: Eastman AQ Branched Polyesters
Appendix C: Eastman AQ Polymers
Appendix D: Eastman AQ 55S and Eastman AQ 38S

***Eastman AQ* Branched Polyesters**

New Hot-Melt Adhesive Raw Materials That Are Water-Dispersible

Eastman AQ branched sulfopolyesters are unique hot-melt adhesive raw materials that are water-dispersible and are useful as base or modifying polymers in hot-melt applications. Recycling processes increasingly require substrates to be bonded with hot-melt adhesives that will separate readily in an aqueous environment. Polyolefin-based adhesives, especially polyethylene and amorphous polypropylene systems, are separable only by physical means due to their low densities compared to water.

There is also a need for hot-melt adhesives that will disperse in an aqueous environment. Eastman Chemical Company's proprietary sulfopolyesters are water-dispersible and applicable to a variety of applications, including packaging and nonwovens. The primary feature of *Eastman AQ* branched polyesters is their

dispersibility in water, coupled with inherent nondispersibility in ionic (saline) solutions, while maintaining good adhesive performance characteristics. These amorphous copolyesters are thermoplastic, rather than thermosetting, and are, therefore, remelttable, which results in their utility for heat-sealable substrate bonding. Eastman's commercial product line offers adhesive formulators a range of melt viscosities from 4,500–400,000 cP. *Eastman AQ* branched polyesters are compatible with a variety of hydrophobic polar ingredients, such as tackifiers, resins, extending oils, and plasticizers, for formulating water-dispersible adhesives. These finished adhesives are then rendered water-dispersible by the efficacy of the sulfopolyester as a surfactant.

Key Benefits

- Easily formulated with a variety of tackifiers, waxes, and plasticizers to meet customer needs
- 100% water-dispersible
- Repulpable under neutral and alkaline conditions
- Wide range of viscosities
- Compatibility with polar ingredients
- Regulated by FDA at 21 CFR 175.105 for use in food-packaging adhesives
- Excellent adhesion to polyolefin substrates
- Nonirritant
- Nondispersible in ionic solutions
- Commercially available
- On TSCA inventory

Table 1
Physical Properties

Typical Properties*	Production Status/Name			
	Commercial AQ 1045	Commercial AQ 1350	Commercial AQ 1950	Commercial AQ 14000
Brookfield Thermosel viscosity @ 177°C, cP (mPa·s)	4,500	35,000	95,000	400,000
Gardner color (molten), max.	4	4	4	4
Physical form	Clear solid	Clear solid	Clear solid	Clear solid
Ring and ball softening point, °C (ASTM E 28)	85	105	115	133
Penetration hardness, dmm (ASTM D 5)	30	14	8	7
T _g (DSC), °C (ASTM D 3418)	-5	-2	3	7
Tensile strength, MPa (ASTM D 412)	—	0.27	0.38	0.61
Elongation, %	1,660	1,600	1,400	1,200
Hydroxyl number	28	28	28	28

*Based on preliminary testing. Properties are typical of average lots. Eastman makes no representation that the properties in any given lot will conform exactly to the listed values.

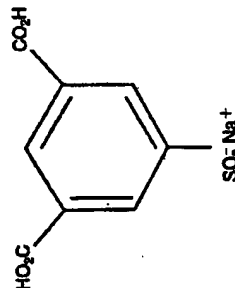
*Brookfield Thermosel viscosity RVDV1A, 10 g of each sample conditioned at 80°C for 16 h in a vacuum oven prior to testing

Technical Aspects of Polyesters

Eastman AQ water-dispersible polyesters are branched, amorphous materials composed of difunctional acids and various glycols. Their water-dispersibility is due to the presence of

pendant sodiumsulfate groups randomly distributed along the polymer backbone. Incorporation of the ionic moieties is readily accomplished by the copolymerization of 5-sodiumsulfisophthalic acid (see Figure 1) into the polymer backbone.

Figure 1
5-Sodiumsulfisophthalic Acid



The unique properties of the new polyesters stem from a branched architecture and a specific combination of monomers, resulting in a low T_g and enhanced compatibility with other resins. The branched structure of the polyesters results in low melt viscosities required for hot-melt adhesive formulations.

Applications

Case/Cartron Closing—Using the key criteria of low viscosity and fast set time, a starting-point formulation using *Eastman AQ 1045* polyester as the base polymer was identified. It is compared to conventional EVA- and PE-based adhesives in Table 2. The water-dispersible polyester-based formulation compared favorably to the performance characteristics of the conventional adhesives for elevated-temperature performance and setting characteristics. As noted, *Eastman AQ 1045* is 100% repulpable in neutral or alkaline conditions.

Multiwall Bags—A formulation using *Eastman AQ 1950* polyester as the base material was identified for use in multiwall bag end seam applications. This formulation is compared to a typical PE-based adhesive in Table 3. Again, the polyester-based composition compared favorably to the conventional adhesive in elevated-temperature performance, yet it is fully repulpable under alkaline conditions. This makes the polyester-based formulation ideally suited for such applications as beater bags.

PET Bottle Label Adhesive—Formulations using *Eastman AQ 1045* polyester should find use for PET bottle labeling. A formulation with many desirable properties for a label adhesive is shown in Table 4. This formulation demonstrates excellent adhesion to PET and good bond strength at 120°F and at 35°F, low ring and ball softening point, and low viscosity at application temperature. This formulation is also completely dispersible in neutral and alkaline repulping conditions.

Nonwoven Product Assembly Adhesive—*Eastman AQ 1350* is beneficial in nonwoven applications. A starting formulation is shown in Table 5. The polyester formulation has excellent adhesion to polyethylene films, *Brookfield Thermosel* viscosity between 1,000–2,000 cP at 177°C, and a low ring and ball softening point of 77°C. Because of the polyester's nondispersibility in ionic solutions such as body fluids, it should provide superior performance in nonwoven applications. This formulation is fully repulpable in alkaline conditions.

Pressure-Sensitive Adhesives—*Eastman AQ* water-dispersible polyesters should also benefit pressure-sensitive adhesive applications. As shown in Table 6, they can be modified to exhibit high levels of tack and moderate holding power. These formulas also have good adhesion to polyethylene, polypropylene, and exceptional adhesion to PET. They also exhibit fiber-tearing quick tack to paper substrates. In repulpability tests, the two-component combination of *Eastman AQ 1350* and *BenzoFlex 9-88* was completely repulpable in all conditions; the three-component formula using *Eastman AQ 14000*, *Staybelite* resin, and *BenzoFlex 9-88* was 70% repulpable in neutral conditions and completely repulpable in alkaline conditions.

Additional pressure-sensitive adhesive applications based on these branched sulfopolyesters include medical tapes that are removable by water but are resistant to ionic fluid such as perspiration.

Table 2

Eastman AQ Formulations

Eastman AQ Formulations		EVA- and PE-Based Hot Melts	
	Wt %		Wt %
Eastman AQ 1045	60.0	Typical EVA	100
Neveex 100	31.0	Typical PE	100
Benzoflex 352	8.7		
Irganox 1010	0.1		
Cyanox 1212	0.2		
Viscosity @ 177°C, cP (ASTM D 3238)	1,125		900
Set time, s	3.1		1.7
SAFT, °C	75		96
PAFT, °C	40		58
Repulpability, alkaline conditions, %	100	NA	NA
RBSPT, °C (ASTM E 28)	84	112	108

*70% pH of 11

Figure 2

Thermal Stability of Eastman AQ

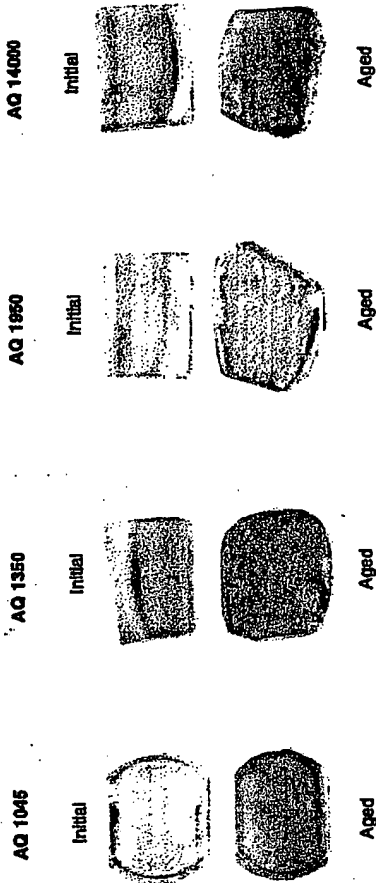


Figure 3

Ionic Stability of Eastman AQ

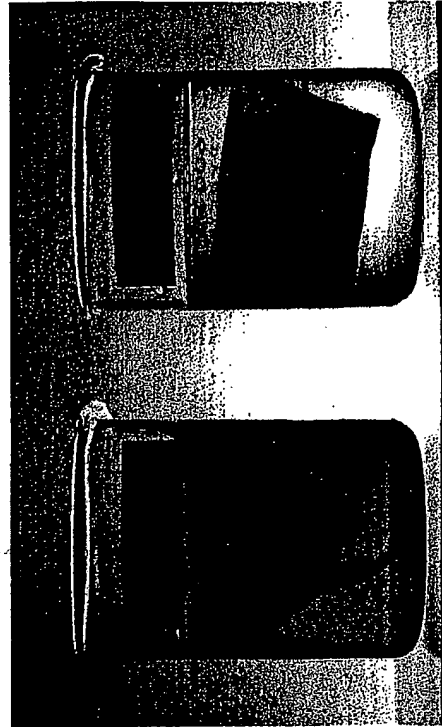


Figure 2 indicates thermal color stability of each *Eastman AQ* branched polyester after samples were aged at 80°C for 100 hours in a forced-air oven. This photo shows the excellent thermal stability of these polyesters. *Eastman AQ* branched polyesters contain both primary and secondary antioxidants.

Figure 3 shows the resistance of *Eastman AQ* 1350 polyester to ionic solutions. This photo depicts two beakers, one filled with tap water, the other filled with 0.2-M ionic solution similar to human body fluids. Dyed *Eastman AQ* 1350 laminated onto nonwoven material was placed in each beaker for 8 hours. The tap water readily dispersed the polyester, while in the ionic solution the AQ 1350 remained nondispersible.

Table 3

Hot Melt Multiwall Bag Adhesive Formulations

Eastman AQ Formulation		PE-Based Hot Melt	
	Wt %		Wt %
Eastman AQ 1960	70.0	Typical PE	—
Nevex 100	20.0		100
Benzoflex 352	9.7		—
Irganox 1010	0.3		—
Viscosity @ 177°C, cP	18,450		32,100
RBSP, °C	102		108
SAFT, °C	85		99
PAFT, °C	60		68
Repeatability, alkaline conditions, %	100		NA

*73°F, pH of 11

Table 4

PET Label Adhesive Formulation Using Eastman AQ 1045

Ingredients		Wt %
Eastman AQ 1045		60.0
Stybelite acid rosin tackifier		26.7
Benzoflex 9-88 plasticizer		13.0
Irganox 1010 antioxidant		0.3
Test Properties		
180° peel, steel, g/mm		38
T-peel adhesion to PET, g/mm		42
90° quick tack, g/mm		15
Room temp hold power, 1 kg, 1 in. ² , 25°C, min		4
Ring and ball softening point, °C		62
Brookfield Thermosel viscosity, 177°C, cP		430
Repeatability, neutral conditions, %		100
Repeatability, alkaline conditions, %		100

*73°F, pH of 7

*73°F, pH of 11

Table 5

Nonwoven Product Assembly Adhesive Using Eastman AQ 1045

Ingredients		Wt %
Eastman AQ 1350		60.0
Stybelite acid rosin tackifier		34.7
Benzoflex 9-88 plasticizer		5.0
Irganox 1010 antioxidant		0.3
Test Properties		
180° peel adhesion, g/mm		60
T-peel adhesion to PE, g/mm		42
90° quick tack, g/mm		1.3
Room temp hold power, 1 kg, 1 in. ² , 25°C, min		1,300
Ring and ball softening point, °C		77
Brookfield Thermosel viscosity, 177°C, cP		1,120
Repeatability, alkaline conditions, %		100

*73°F, pH of 11

Eastman Chemicals

EASTMAN AQ® POLYMERS **Properties and Applications**

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EASTMAN AQ polymers are well suited for a variety of applications, including adhesion, bonding, dispersion, coating, priming, and flocculating.

$$\text{HO}-\text{G}-\text{A}-\text{G}-\underset{\substack{| \\ \text{SO}_3^- \text{Na}^+}}{\text{A}}-\text{G}-\text{A}-\text{G}-\underset{\substack{| \\ \text{SO}_3^- \text{Na}^+}}{\text{A}}-\text{G}-\text{OH}$$

-OH = hydroxy end groups

EASTMAN AQ polymer types differ chiefly in glass transition temperature (T_g) or softening point. The "dry" T_g of each polymer is indicated by its numerical designation. For example, the dry T_g of EASTMAN AQ 29 polymer is 29°C. The "S" or "D" which follows the T_g refers to the solid or dispersed form of the polymer, both of which are available commercially.

- Are amorphous, thermoplastic polyesters
- Adhere well to a variety of substrates
- Provide heat sealable bonding
- Provide fast setting speeds
- Are inherently dispersible in water
- Will disperse certain hydrophobic materials (organic liquids, oils, tackifiers and waxes) in water and dissolve certain water-insoluble organics
- Are miscible with many waterborne polymer emulsions and latexes
- Form hard, clear films without need for solvents
- Are safe, easy to handle, and easy to clean up
- Are supplied and serviced by EASTMAN

TYPICAL PROPERTIES OF EASTMAN AQ® POLYMERS^a

	Solid Polymers		
	AQ 29S	AQ 38S	AQ 55S
Percent solids	100	100	100
Physical form	Pellet	Pellet	Pellet
Approximate molecular weight, Mn	16,000	14,000	14,000
Hydroxyl number	<10	<10	<10
Acid number	<2	<2	<2
T _g , °C	29	38	55
Melt viscosity at 200°C (392°F), poise ^b	2000	9700	42,000
Color	Clear, light amber	Clear, light amber	Clear, light amber
	Dispersed Polymers		
	AQ 29D	AQ 38D	AQ 55D
Percent solids (in water)	30	25	28
Ionic type	Anionic	Anionic	Anionic
pH	5-8	5-6	5-6
Viscosity, cP at 100 rpm ^c	45	30	42
Density at 24°C (75°F)			
lbs/gal	8.83	8.84	8.97
g/mL	1.08	1.06	1.08
COD, mg/g solids	1630	1700	1750

^a These properties are typical of average lots. Eastman makes no representation that the material in any particular shipment will conform to the values given.

^b Seglar-McKevrey Capillary Rheometer, 100 sec⁻¹ shear rate.

^c Brookfield Viscometer, Model RVT, Spindle No. 1, 25°C (77°F).

NOTE: Tests conducted at an independent laboratory indicate that these polymers are not readily biodegradable. BOD test results depend on many variables. Biodegradability of individual organic compounds depends to a large extent upon the individual wastewater treatment system biological community. Thus, individual testing of specific waste streams would be needed to obtain biodegradability data applicable to specific situations.

Dispersions of EASTMAN AQ Polymers are:

- Solvent-free
- Surfactant-free
- Freeze-thaw stable^a
- Water-reducible
- Viscosity-adjustable with thickeners
- Compatible with nonionics and anionics
- Incompatible with cationics

^a Can be readily regenerated if frozen.

APPLICATIONS

WATERBORNE ADHESIVES

Cold-Glue Bonding

EASTMAN AQ[®] 29 polymer and, to a lesser extent, *EASTMAN AQ* 38 and *EASTMAN AQ* 55 polymers exhibit good adhesion to a number of important substrates, such as PET plastics, metals, wood and glass. *EASTMAN AQ* polymers are well suited for liquid adhesive or cold-glue bonding applications that do not require a high degree of water or moisture resistance.

EASTMAN AQ 29 Polymer Adhesion to Various Substrates^a

Good	Fair	Poor
Aluminum Foil	<i>Lucite</i> Acrylic Sheet	Cellulose Acetate/Butyrate
Cellulose Acetate	Particle Board	Polyethylene
Cellophane	Polystyrene	Polypropylene
Cold-rolled Steel	<i>Saran</i> Wrap	Plasticized PVC (Vinyl)
Galvanized Steel		
Glass		
<i>KODAR</i> [®] PETG Film		
Leather		
<i>Lexan</i> Polycarbonate		
Paper		
PET Film		
Wood		

^aUsing Test Method ASTM D 3808.

When *EASTMAN AQ* polymers are used in cold-glue bonding, at least one of the substrates must be water-absorptive. If this condition is met, *EASTMAN AQ* polymers will produce substrate-tearing bonds with many substrate pairs. The following are examples of substrate pairs bonded with *EASTMAN AQ* 29D polymer (30% solids) that resulted in substrate, rather than adhesive, failure.

Bonded Pairs Resulting in Substrate Failure

Substrate 1	Substrate 2	Substrate 1	Substrate 2
Cardboard	Cardboard	Aluminum Foil	Wallboard
Carton Stock	Carton Stock	Kraft Paper	Cold-rolled Steel
Cellophane	Carton Stock	Kraft Paper	Styrofoam
OPET ^a Film	Copier Paper	Kraft Paper	Particle Board
Kraft Paper	Glass	Kraft Paper	Polyurethane Foam
Rag Bond Paper	Glass		

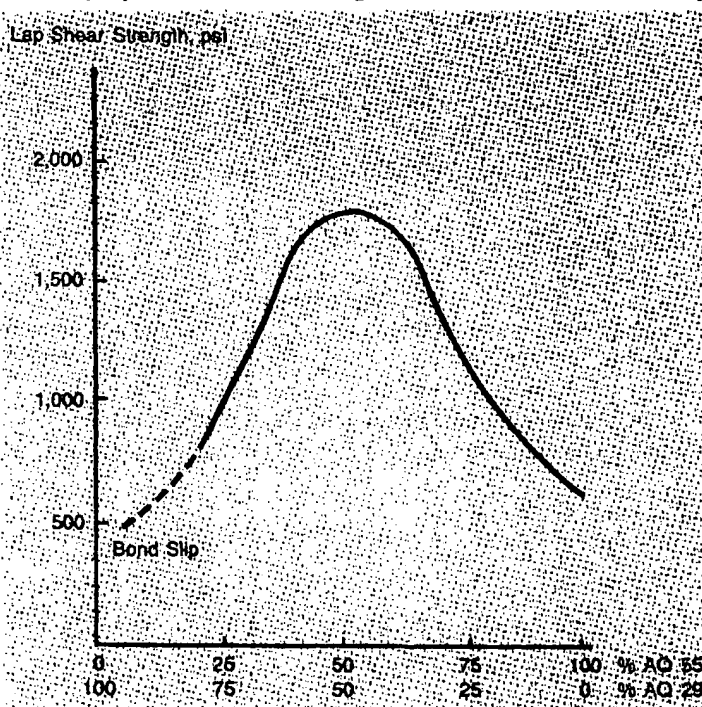
^aOriented Polyethylene Terephthalate

EASTMAN AQ polymer dispersions lose water rapidly on absorptive substrates, allowing surprisingly fast setting speeds. For instance, in bonding kraft paper to rag bond paper, the setting speed for *EASTMAN AQ* 29D polymer, at 30% solids, is in the 9- to 12-second range, as measured by TAPPI Method UM 646. This is in the same time range as a conventional vinyl acetate/ethylene (VAE) copolymer emulsion at 55% solids.

Wood Bonding

Even though *EASTMAN AQ*® polymers exhibit good adhesion to hard and soft woods, none of the *EASTMAN AQ* polymers alone forms strong, fiber-tearing wood bonds. For bonds conditioned at 50% relative humidity, *EASTMAN AQ 29* polymer is too flexible and *EASTMAN AQ 55* polymer is too brittle to produce fiber tear. However, blends of *EASTMAN AQ 29* and *EASTMAN AQ 55* polymers form stronger bonds than either one alone, or *EASTMAN AQ 38* polymer alone. This blend produces slight-to-moderate fiber tear on hard maple wood in the lap shear test (ASTM D-1002). Maximum bond strength is achieved at about a 50:50 blend level.

Effect of *EASTMAN AQ 29* and *EASTMAN AQ 55* Polymer Blend Ratios on (Hard Maple) Wood Bond Strength at 50% Relative Humidity



Still stronger bonds with deeper fiber tear are obtained by adding a polyvinyl acetate (PVAc) latex to the 50:50 blend of *EASTMAN AQ 29D* and *EASTMAN AQ 55D* polymers. This addition imparts good working properties to the adhesive, including fast wood wet-out, easy bond alignment, low buildup on sanding belts, and easy cleanup.

Suggested Starting Formulation for Wood Adhesives

<i>EASTMAN AQ 29D</i> Polymer (30% solids)	1.00 part
<i>EASTMAN AQ 55D</i> Polymer (28% solids)	1.00 part
Acrysol ICS-1 Acrylic Thickener (30% solids)	0.025 part
Add dilute (5%-10%) Ammonia Solution to pH 8.	

HEAT-SEALING ADHESIVES

Paper Bonding

The thermoplastic nature of *EASTMAN AQ*[®] polymers enables them to be used as heat-sealing adhesives for some important substrates. *EASTMAN AQ* polymers produce paper-tearing bonds at relatively low temperatures, as shown below. Minimum bonding temperature increases, predictably, as T_g and melt viscosity increase.

Minimum Heat Sealing Temperature [°C (°F)] of *EASTMAN AQ* Polymers^a

<i>EASTMAN AQ</i> 29	79 (175)
<i>EASTMAN AQ</i> 38	93 (200)
<i>EASTMAN AQ</i> 55	107 (225)

^aMinimum temperature required to produce fiber-tearing bonds on 50% rag bond paper. (Sentinel Heat Sealer, Model 12 ASL, 3 sec/40 psi).

Aluminum and OPET Bonding

Temperatures well above the minimum paper bonding temperatures are required to bond aluminum foil and OPET (oriented polyethylene terephthalate) film to themselves or to each other. For sealing with *EASTMAN AQ* 29 polymer, the best results have been obtained in the 149°-163°C (300°-325°F) range. Higher temperatures are required to bond with *EASTMAN AQ* 38 and *EASTMAN AQ* 55 polymers. In practice, higher machine temperatures and much shorter contact times will probably achieve the same result as lower machine temperatures and longer contact times.

Peel Strength of Bonds Formed by Heat Sealing with *EASTMAN AQ* 29 Polymer^a

Substrate No. 1	Substrate No. 2	Film Thickness, mils	Peel Strength, g/in.	
			Maximum	Average
Coated OPET	Coated OPET	1.0	1300	550
Coated OPET	Coated OPET	0.5	680	450
Coated OPET	Uncoated OPET	1.0	400	200
Coated Aluminum	Coated Aluminum	1.0	1300	820
Coated Aluminum	Uncoated Aluminum	1.0	620	450
Coated OPET	Uncoated Aluminum	1.0	900	630
Coated Aluminum	Uncoated OPET	1.0	430	270

^aApproximate adhesive T-Peel Strength using Test Method ASTM D-1876-72 and Sentinel Heat Sealer, Model 12 ASL, 107°C (325°F)/3 sec/40 psi (Heated on both sides).

Stronger bonds can be obtained by precoating both substrates with a thin film of *EASTMAN AQ* 29 polymer. When precoating only one substrate, applying *EASTMAN AQ* 29 polymer to the OPET substrate will produce stronger bonds than if the adhesive precoat is applied to the other substrate.

DISSOLUTION AND DISPERSION OF HYDROPHOBIC MATERIALS

EASTMAN AQ® polymers have the capacity to dissolve or disperse many water-insoluble, hydrophobic materials in water. This property is especially useful in applications in which the exclusion of solvents and conventional surfactants is desirable, such as preparing water-based adhesives, coatings, emulsions, and primers. **EASTMAN AQ** polymers can be used to disperse (or in some cases to solubilize) hydrophobic organic materials in water.

Dissolution

Many organic compounds of low water solubility are miscible (soluble) to a degree in **EASTMAN AQ** polymer dispersions. For example, 10 parts of toluene will dissolve^a in 100 parts of **EASTMAN AQ 55D** polymer with only moderate agitation. There are, of course, limits to the amount of a given organic material that can be absorbed by an **EASTMAN AQ** polymer dispersion. The limit for toluene appears to be about 20 parts per 100 parts of **EASTMAN AQ 55D** polymer. At this level of toluene, the **EASTMAN AQ** polymer dispersion becomes cloudy and highly viscous. Some materials, such as chlorobenzene, cause the **EASTMAN AQ** polymer dispersion to gel at higher organic levels.

^aThe term dissolve is used here to indicate that the organic phase is absorbed by the aqueous **EASTMAN AQ** polymer dispersion with little or no change in the appearance of the dispersion.

Dissolution of Selected Organic Solvents and Plasticizers in **EASTMAN AQ 55D** Polymer Dispersions

Organic Solvent/Plasticizer	10 phr	20 phr
Chlorobenzene	M	M*
Cyclohexane	I	I
Dibutyl Phthalate	P	P
p-Diisopropylbenzene	I	I
Diisopropyl Ketone	I	I
2,5-Dimethyl-2,4-hexadiene	I	I
Dimethyl Phthalate	M	M*
Dioctyl Adipate	I	I
Ethylbenzene	P	P
2-Ethylhexanol	I	I
Hexane	I	I
1,5-Hexanediol	M	M
Methyl-n-amyketone	M	P
Methyl Benzate	M	M
Methylene Chloride	M	M
Methyl Salicylate	M	M
TELAND® Ester Alcohol	I	I
Toluene	M	P
Xylene	P	P

U = Miscible; P = Partially Miscible; I = Immiscible; * = Gelled

In dispersions of **EASTMAN AQ 55** polymer, aliphatic and cycloaliphatic hydrocarbons are immiscible; aromatic hydrocarbons are generally miscible or partially miscible. However, if the aliphatic fraction of the molecule is too high, as with diisopropyl benzene, the aromatic hydrocarbon is immiscible.

Dispersion

Certain hydrophobic materials that are not miscible in *EASTMAN AQ*[®] polymer dispersions can be incorporated by using a high-shear mixer such as a Kady mill or homogenizer. High loadings (up to 100 parts hydrophobe:1 part *EASTMAN AQ* polymer solids, in some cases) of the hydrophobe can be achieved; however, these dispersions may show some separation after a few days.

In general, 80 parts of oil or 10 to 15 parts of wax can be dispersed by one part of *EASTMAN AQ* polymer at 50% total solids. In some instances, ratios as high as 100:1 (hydrophobe:*EASTMAN AQ* polymer) have been achieved. Once formed, dispersions of hydrophobe:*EASTMAN AQ* polymer are water-reducible. They are also miscible with many latex adhesive products, thus providing a convenient means for introducing plasticizers, tackifiers, and other water-immiscible materials into latex systems without additional surfactants.

Hydrophobes that are liquid at room temperature, such as silicone oil, can be incorporated, with high-shear mixing, directly into *EASTMAN AQ* polymer dispersions; solids that melt at a low temperature, such as paraffin, can be incorporated, again with high-shear mixing, by pouring molten material into *EASTMAN AQ* polymer dispersions heated to about 80°C (176°F).

All three *EASTMAN AQ* polymers exhibit the capacity to disperse hydrophobes to some degree; however, *EASTMAN AQ* 55 polymer is generally a more effective dispersing agent than either *EASTMAN AQ* 29 or *EASTMAN AQ* 38 polymer.

Procedures for dispersing hydrophobes are found in the Procedures Section.

ENCAPSULATION

Certain naturally occurring oils and fragrances are also miscible with *EASTMAN AQ* polymer dispersions. This feature can be used to good advantage to encapsulate fragrances, disinfectants, fungicides, and insect repellents in *EASTMAN AQ* polymer films and powders for slow release. Pine oil, lemon oil, citronella oil and *Aramis* Fragrance No. K-1167 are examples of materials that have been successfully encapsulated in *EASTMAN AQ* polymer films (cast or sprayed) and powders (freeze dried or spray dried).

Other Potential Applications:

- Base for household adhesives
- Dispersant for pigments and water-insoluble dyes
- Primer or tie-coat for wood finishes
- Primer for PET film coatings
- Temporary, water-removable protective coatings for metal, plastic, wood, and glass
- Flocculating agent for wastewater sedimentation
- Dust suppressant

CHARACTERISTICS

GLASS TRANSITION TEMPERATURES

The glass transition temperatures (T_g) of *EASTMAN AQ*® polymers range from approximately 29°C to 55°C. T_g is determined largely by the choice of glycols used to make the polymer.

MOISTURE REGAIN

Because of their ionic nature and the presence of hydrophilic glycols, *EASTMAN AQ* polymers absorb considerable moisture from ambient air. Moisture regain lowers the T_g of *EASTMAN AQ* polymers.

Effect of Moisture Regain on T_g of *EASTMAN AQ* 29 Polymer^a

Relative Humidity, %	Moisture Regain, %	T_g , °C
Dry	—	29
65	2.0	19
78	4.0	13
85	5.6	8

^aTwo-week conditioning at 21°-23°C (70°-73°F) of 3 mil film.

Moisture uptake also affects the tensile properties of *EASTMAN AQ* polymers in a predictable way: as moisture content increases, tensile breaking strength and initial modulus decrease, and elongation at break increases.

Effect of Moisture Regain on Tensile Properties of *EASTMAN AQ* 29 Polymer

Property	Relative Humidity	
	Dry	65%
Breaking strength, psi	1500	380
Initial modulus, 10 ³ psi	60	1.4
Elongation at break, %	460	900

MORPHOLOGY

EASTMAN AQ polymers are thermoplastic and amorphous; they show no detectable crystal melting by differential scanning calorimetry.

END GROUPS

Most of the end groups of *EASTMAN AQ* polymer chains are hydroxy groups. Although hydroxyl numbers are fairly low, typically less than 10, the potential for cross-linking reactions still exists, such as reaction with melamine/formaldehyde resins, and for chain extension with, for example, diisocyanates. The carboxyl (—COOH) content of the *EASTMAN AQ* polymers is low but detectable; acid numbers are typically less than two.

MELT VISCOSITIES

The melt viscosities of *EASTMAN AQ*® polymers are much higher than those of common hot-melt adhesives such as polyolefin and EVA (ethylene/vinyl acetate copolymer). These high melt viscosities, along with the sensitivity of polyesters to hydrolytic breakdown at elevated temperatures, preclude the use of *EASTMAN AQ* polymers in conventional hot-melt adhesive applicators and reservoirs. However, thin films and powders can be melted for a few seconds — long enough to form a bond — without significant degradation.

SOLUBILITY

EASTMAN AQ polymers (100% solids) are soluble^a in certain solvents and solvent mixtures at room temperature.

Solvent	AQ 295	AQ 385	AQ 555	Solvent	AQ 295	AQ 385	AQ 555
Esters				Chlorinated Solvents			
Ethyl Acetate	SW	SW	SW	Methylene Chloride	S ^{sv}	S ^{sv}	S ^{sv}
Methyl Acetate	SW	SW	SW	General Solvents			
EKTASOLVE® PM Acetate	SW	I	I	N,N-dimethylformamide	S	S	S
n-Butyl Acetate	I	I	I	Dimethyl Sulfoxide	S	S	H
EKTASOLVE EB Acetate	I	I	I	Tetrahydrofuran	H ^v	H ^v	S
Alcohols				2-Nitropropane	SW	SW	I
Ethyl Alcohol (anhyd)	I	I	I	EKTAPRO® EEP Solvent	SW	SW	I
Ethyl Alcohol (95%)	SW	I	I	Diethylene Glycol	S ^{sv}	S ^{sv}	S ^{sv}
Isopropyl Alcohol	I	I	I	Propylene Glycol	H	I	I
Methyl Alcohol	SW	SW	SW	Mixtures			
TEXANOL® Ester Alcohol	I ^{sv}	I ^{sv}	I	Cyclohexanone:			
Glycol Ethers				Methyl Alcohol, 8:1	S	S	S
EKTASOLVE DM Solvent	H	S	H	Methylene Chloride:			
EKTASOLVE EB Solvent	I	I	I	Methyl Alcohol, 8:1	S	S	H
Hydrocarbons				Tetrahydrofuran:			
Hexane	I	I	I	Methyl Alcohol, 8:1	S	S	S
Toluene	SW	SW	I	Toluene:Ethyl:			
Ketones				Alcohol (anhyd), 8:1	SW	SW	SW
Acetone	SW	SW	SW				
Methyl Ethyl Ketone	SW	SW	SW				
Cyclohexanone	S ^v	S ^v	S				

H — Dispersed (ail), hazy solution

I — Insoluble; nearly unaffected

S — Completely soluble, clear solution

SW — Pellets swollen, not soluble

sv — Slightly viscous

v — Viscous

hv — Highly viscous

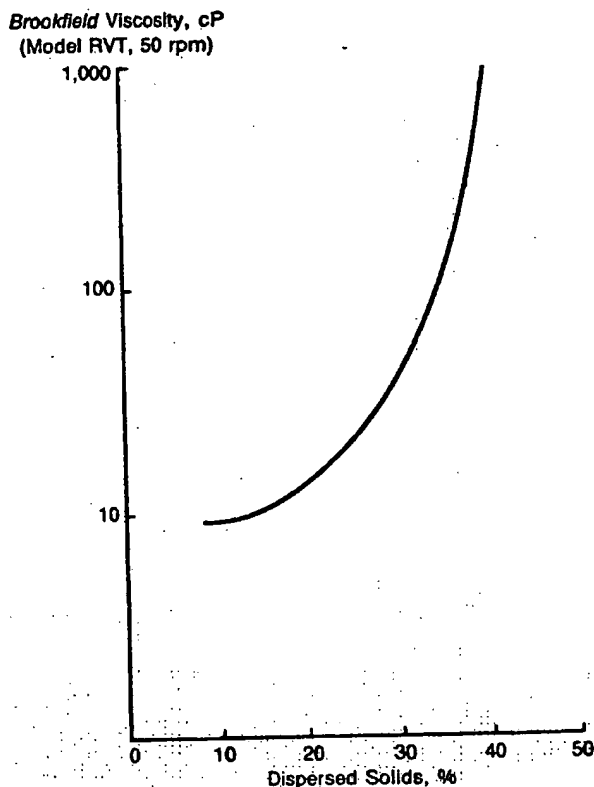
^a Soluble — one gram polymer pellets dissolved in nine grams of solvent (agitated) within one week at room temperature (~22°C (72°F)).

AQUEOUS DISPERSIONS OF EASTMAN AQ® POLYMERS

VISCOSITY OF PURE POLYMER DISPERSIONS

Pellets of *EASTMAN AQ* polymers disperse directly in hot, soft water without the assistance of cosolvents, surfactants, neutralizing amines, or neutralizing alkalies. Only moderate agitation is needed — just enough to keep the pellets in motion. Complete dispersion is usually achieved in 20 to 30 minutes. Dispersions of 30% solids (nonvolatile matter) or less are very low in viscosity (<50 cP). As the solids level increases above 30%, the dispersion viscosity increases rapidly. Forty percent solids is near the maximum level that can be achieved for *EASTMAN AQ 29* polymer. Attempts to disperse beyond 40% solids result in gels. *EASTMAN AQ 38* and *EASTMAN AQ 55* polymers behave similarly but have somewhat lower maximum levels.

Viscosity as a Function of Dispersed Solids for
EASTMAN AQ 29 Polymer



Soft or demineralized water (total hardness less than 20 ppm) should be used in combination with, and to disperse, *EASTMAN AQ* polymers. Hard water ions (Ca^{+2} , Mg^{+2} , Fe^{+3} , etc.) will retard the dispersion rate of *EASTMAN AQ* polymers in water and, if present in sufficient concentration, will produce opaque and even unstable dispersions. High concentrations of electrolytes should be avoided; even monovalent electrolytes such as sodium chloride can cause coagulation or gelling of *EASTMAN AQ* polymer dispersions.

THICKENING POLYMER DISPERSIONS

The viscosity of EASTMAN AQ® polymer dispersions can be increased by the addition of conventional thickening agents, such as those listed below.

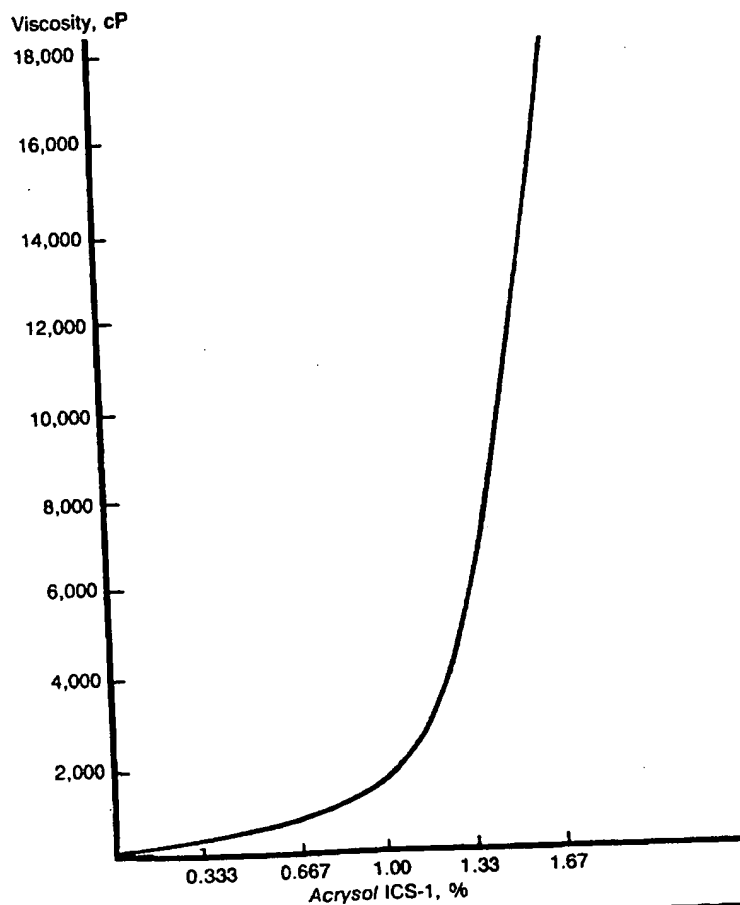
Thickeners for EASTMAN AQ Polymer Dispersions

Acrysol ICS-1 — Rohm and Haas Company
Cab-O-Sil M5 Colloidal Silica — Cabot Corporation
Kelzan S Xanthan Gum — Kelco
Starch and Modified Starches
Vinol 540 Partially-Hydrolyzed Polyvinyl Alcohol^a — Air Products

^aMedium- and fully-hydrolyzed polyvinyl alcohols are not compatible.

The viscosity of EASTMAN AQ 29D polymer (30% solids) is shown below as a function of Acrysol ICS-1 concentration.

Viscosity of EASTMAN AQ 29D Polymer Thickened with Acrysol ICS-1 and Dilute Ammonium Hydroxide to pH 8



FILM FORMING, PLASTICIZATION, AND CROSS-LINKING

Unlike many latex materials, dispersions of *EASTMAN AQ*[®] polymers have little tendency to "skin" or dry out in open containers. The low-skinning tendency and the fast redispersibility of *EASTMAN AQ* polymers in warm water make cleanup of equipment such as application wheels and spray nozzles relatively easy and quick.

To produce coherent films that adhere well to many substrates, dispersions of *EASTMAN AQ* 29 and *EASTMAN AQ* 38 polymers may be evaporated to dryness at room temperature. To obtain coherent films of *EASTMAN AQ* 55 polymer at room temperature, a small amount (3%-5% based on dry weight) of a plasticizer is helpful. Film flexibility and adhesion depend on polymer T_g , moisture content and plasticizer level, if any. In the absence of cross-linking agents, *EASTMAN AQ* polymer films will redisperse in hot water, even after drying at $>100^{\circ}\text{C}$ (212°F).

The flexibility of *EASTMAN AQ* polymer films is readily increased by dissolving water-soluble plasticizers in the dispersion before the film is formed. Many water-soluble glycols and polyols can be used for this purpose. Examples include ethylene glycol, diethylene glycol, polyethylene glycols, glycerol, and sorbitol. Film flexibility, softness, tackiness, and water sensitivity generally increase as the level of plasticizer increases.

Although the level of reactive functional groups in *EASTMAN AQ* polymers is low, stronger and more water-resistant films are produced by formulating *EASTMAN AQ* polymers with certain melamine/formaldehyde cross-linking resins and an appropriate catalyst. After drying, a thermal treatment is usually necessary to obtain a sufficient degree of curing (cross-linking). Examples of cross-linking formulas are given below. Film stiffness increases as the level of cross-linking resin in the formula increases.

Examples of Curable *EASTMAN AQ* Polymer Formulations

	Total Parts	
	3:1 ^a	5:1 ^a
<i>EASTMAN AQ</i> 29D Polymer (30% solids)	72.0	80.0
<i>Cymel</i> 303 Resin (100% solids)	7.2	4.8
<i>Igepal</i> CO 977 Nonionic Stabilizer (70% solids)	1.7	1.7
Add Water to 30% solids	19.1	13.5

^aRatio of *EASTMAN AQ* polymer to cross-linking resin (solids basis).

The water resistance of *EASTMAN AQ* polymer films increases as T_g increases. Films of *EASTMAN AQ* 29 polymer disperse in water at room temperature in a matter of a few minutes; *EASTMAN AQ* 55 polymer films, on the other hand, swell in water at room temperature but do not disperse. Water resistance of an *EASTMAN AQ* polymer film can be increased by baking the film to a "bone dry" condition or by rubbing the surface with a solution of a polyvalent metal salt, such as dilute aluminum sulfate solution.

EFFECT OF pH ON DISPERSION STABILITY

Dispersions of *EASTMAN AQ*[®] polymers in water are most stable at pH 5-7. In this pH range, there is no significant reduction in polymer molecular weight, even after one year of storage at room temperature [20°-23°C (68°-73°F)]. Below pH 5, acid-catalyzed hydrolysis results in molecular weight breakdown, especially at elevated temperatures. Likewise, above pH 7, saponification of the polyester becomes operative and can result in precipitation of a nondispersible residue. Both acidic and basic degradation reactions are accelerated by increasing temperature and by increasing acid or base concentration.

EFFECT OF SOLVENTS ON DISPERSING STABILITY

Many organic solvents, even those having low water solubility, are miscible to the extent of ten weight percent or more in *EASTMAN AQ* polymer dispersions. The effect of several common solvents on the stability and viscosity of *EASTMAN AQ 29D* and *EASTMAN AQ 55D* polymer dispersions is shown below. In most cases, little or no separation or change in viscosity is observed after 30 days.

Effect of Solvents on Dispersion Stability

Solvent ^a	<i>EASTMAN AQ 29D</i> Polymer				<i>EASTMAN AQ 55D</i> Polymer			
	Viscosity, cP ^b			Condition ^c	Viscosity, cP ^b			Condition ^c
	Initial	7 days	30 days		Initial	7 days	30 days	
Acetone	31	31	31	M	29	29	29	M
Ethyl Acetate	56	56	62	M	59	51	49	M
Ethyl Alcohol	33	32	32	M	31	32	32	M
Methyl Ethyl Ketone	41	43	42	M	35	36	35	M
i-Propyl Alcohol	35	36	35	M	31	32	32	M
1,1,1-Trichloroethane	44	40	42	S	58	55	50	M
Toluene	49	46	46	M	140	94	80	M

^a 40g solvent mixed with 400g *EASTMAN AQ 29D* or *EASTMAN AQ 55D* polymer dispersion in high-speed mixer for one minute.

^b Brookfield Viscometer, Model RVT, Spindle No. 2, 50 rpm.

^c M = Miscible after 30 days; S = Some separation after 30 days.

COMPATIBILITY^a OF DISPERSIONS

Latexes and Emulsions

EASTMAN AQ[®] polymer dispersions are compatible with many anionic and nonionic polymer latexes and emulsions. Cationics are generally not compatible with EASTMAN AQ polymer dispersions.

^aThe term compatible is used in this section to indicate that the materials can be mixed without causing coagulation or precipitation.

Compatibility of EASTMAN AQ 29D Polymer with Commercial Waterborne Polymers^a

Product	Polymer Type	Solids, %	Viscosity ^b , cP				Stability
			Initial	24 Hr	7 Day	28 Day	
Airflex 400	VAE	55	84	90	106	117	Stable
Airflex 405	VAE	55	31	36	43	37	Stable
Airflex 416	VAE-COOH	55	48	50	57	56	Stable
Airflex 4514	Co-VCI	45	—	—	—	—	Unstable
Duracet HT-2	PVAc	38	322	325	328	328	Stable
Duracet 110	PVAc	55	72	73	77	73	Stable
Flexcryl 1625	Acrylic	69	—	—	—	—	Unstable
Flexcryl 1685	Acrylic	55	28	30	34	33	Stable
Flexbond 150	PVAc	55	33	32	32	30	Stable
Nacrylic X4280	Acrylic	51	34	31	30	31	Stable
Neocryl A-5144	Acrylic	37	23	21	19	19	Stable
Neorez R-9431	Urethane	35	50	47	46	43	Stable
Resyn 25-1025	PVAc	55	78	76	79	76	Stable
Vinac XX-230	PVAc	55	251	256	261	284	Stable
Witcobond W-260	Urethane	35	38	31	29	26	Stable

^aEqual weights of EASTMAN AQ 29D polymer (30% solids) and commercial product mixed slowly with low shear.

^bBrookfield Viscometer, Model RVT, 50 rpm.

Compatibility of EASTMAN AQ 55D Polymer with Commercial Waterborne Polymers^a

Product	Polymer Type	Solids, %	Viscosity ^b , cP				Stability
			Initial	24 Hr	7 Day	28 Day	
Airflex 400	VAE	55	139	140	156	162	Stable
Airflex 405	VAE	55	33	34	38	39	Stable
Airflex 416	VAE-COOH	55	48	46	48	48	Stable
Airflex 4514	Co-VCI	45	240	—	—	—	Unstable
Duracet HT-2	PVAc	38	920	790	912	990	Stable
Duracet 110	PVAc	55	80	82	83	73	Stable
Flexcryl 1625	Acrylic	69	492	—	—	—	Unstable
Flexcryl 1685	Acrylic	55	36	35	40	55	Stable
Flexbond 150	PVAc	55	33	32	31	30	Stable
Nacrylic X4280	Acrylic	51	36	36	36	35	Stable
Neocryl A-5144	Acrylic	37	23	22	21	17	Stable
Neorez R-9431	Urethane	35	48	45	45	44	Stable
Resyn 25-1025	PVAc	55	115	115	171 ^c	125 ^c	Separation
Vinac XX-230	PVAc	55	472	461	485	530	Stable
Witcobond W-260	Urethane	35	28	26	24	23	Stable

^aEqual weights of EASTMAN AQ 55D polymer (28% solids) and commercial product mixed slowly with low shear.

^bBrookfield Viscometer, Model RVT, 50 rpm.

^cPhase separation had occurred at time of viscosity measurement.

Fillers

Many common fillers can be added to *EASTMAN AQ*[®] polymer dispersions without causing coagulation or a significant change in viscosity.

Compatibility of Fillers with *EASTMAN AQ* Polymers

Filler	Loading phr (Dry Basis) ^a	<i>EASTMAN AQ 28D</i>				<i>EASTMAN AQ 55D</i>			
		Viscosity, cP (pH) ^b				Viscosity, cP (pH) ^b			
		Initial	1 Day	7 Day	1 Mo	Initial	1 Day	7 Day	1 Mo
Antimony Oxide	50	35 (6.0)	35 (6.0)	35 (6.1)	35 (6.2)	—	—	—	—
Barytes No. 22 (BaSO ₄)	50	16 (6.2)	17 (6.4)	17 (6.8)	16 (6.7)	—	—	—	—
Hydrated Alumina RH-730	50	41 (6.7)	41 (6.6)	41 (6.9)	40 (6.7)	—	—	—	—
Kadox 515 (ZnO)	50	37 (7.6)	36 (7.3)	35 (7.4)	34 (7.2)	—	—	—	—
Sylold 83 Silica	17	51 (4.5)	80 (4.6)	116 (4.9)	68 (5.0)	—	—	—	—
Ti-Pure R-900 (TiO ₂)	50	34 (6.9)	34 (6.9)	33 (7.0)	32 (6.9)	34 (5.7)	—	35 (6.1)	—
Whiting No. 9 (CaCO ₃)	25	30 (8.2)	—	36 (8.3)	—	27 (7.2)	—	28 (7.7)	—
No. 9	50	28 (7.8)	32 (7.8)	36 (7.9)	33 (7.3)	29 (7.4)	—	30 (7.7)	—
No. 9	75	33 (8.1)	—	39 (8.2)	—	32 (7.4)	—	33 (7.8)	—
No. 9	100	35 (8.2)	—	38 (8.3)	—	33 (7.5)	—	35 (7.8)	—

^a phr = parts filler per 100 parts *EASTMAN AQ* polymer.

^b Value in parentheses is pH.

PROCEDURES

I. Dispersing Hydrophobes in EASTMAN AQ® Polymers

Example A: Disperse a low-melting paraffin wax in EASTMAN AQ 55D polymer
10:1 wax/EASTMAN AQ polymer (60% solids)

Step 1 Mix 100 parts of EASTMAN AQ 55D polymer (28% solids) and 133 parts of demineralized water. Heat the mixture to 80°-85°C (176°-185°F) and pour into a preheated high-shear mixer such as a Kady mill; homogenizer, or kitchen blender.

Step 2 In a separate container, melt 280 parts of Shellwax 100 paraffin by heating it to 60°-72°C (140°-160°F).

Step 3 Turn the high-shear mixer ON and slowly pour the molten paraffin wax into the hot, highly sheared aqueous mixture (from Step 1). Continue high-shear mixing for 10 minutes after addition of the molten wax is complete.

Note: The mixing vessel should be kept closed during Steps 3 and 4 to minimize loss of water by evaporation.

Step 4 Reduce the stirring rate to a low level and allow the dispersion to cool to room temperature.

Step 5 Turn the mixer OFF and transfer the wax dispersion to a closed container for storage.

Summary

Ingredients	Total	Solids
EASTMAN AQ 55D Polymer, parts	100	28
Shellwax 100 Paraffin, parts	280	280
Water, parts	133	—
Totals	513	308
Solids, %		60

Caution: This procedure presents a risk of hot liquid splashes. Operators should wear appropriate protective clothing, including a full face shield and gloves, to cover all exposed skin areas.

Example B: Disperse a high-melting aliphatic hydrocarbon tackifier in *EASTMAN AQ® 55D* polymer

5:1 tackifier/*EASTMAN AQ* polymer (55% solids)

- Step 1 Mix 200 parts of *EASTMAN AQ 55D* polymer (28% solids) and 131 parts of demineralized water. Heat the mixture to about 90°C (194°F) and pour into a preheated high-shear mixer such as a Kady mill, homogenizer, or kitchen blender.
- Step 2 In a separate container, melt 280 parts of *EASTOTAC™* hydrocarbon resin H-130 tackifier by heating it to 180°-190°C (356°-374°F).
- Step 3 Turn the high-shear mixer ON and slowly add, in portions, the molten tackifier to the hot, highly sheared aqueous mixture (from Step 1). Continue high-shear mixing for 20 minutes after addition of the molten wax is complete.

Note: The mixing vessel should be kept closed during Steps 3 and 4 to minimize loss of water by evaporation.

- Step 4 Reduce the stirring rate to a low level and allow the dispersion to cool to room temperature.
- Step 5 Turn the mixer OFF and transfer the tackifier dispersion to a closed container for storage.

Summary

Ingredients	Total	Solids
<i>EASTMAN AQ 55D</i> Polymer, parts	200	56
<i>EASTOTAC</i> Hydrocarbon Resin H-130, parts	280	280
Water, parts	131	—
Totals	611	336
Solids, %		55

Caution: This procedure presents a risk of hot liquid splashes. Operators should wear appropriate protective clothing, including a full face shield and gloves, to cover all exposed skin areas.

Example C: Disperse a silicone oil in *EASTMAN AQ® 55D* polymer
50:1 oil/*EASTMAN AQ* polymer (50% solids)

Step 1 Mix 100 parts of *EASTMAN AQ 55D* polymer (28% solids) and 1356 parts of demineralized water in a high-shear mixer such as a Kady mill or homogenizer.

Step 2 Slowly pour 1400 parts of *Dow Corning 200* silicone oil [250 centistokes (cS) viscosity] into the vortex of the mixture (from Step 1). Continue high-shear mixing for 20 minutes after addition of the oil is complete.

Note: Due to the energy imparted by the mixer, the temperature of the dispersion may rise to 70°-80°C (158°-176°F) during this time. No other heat is applied. Keep the container closed during Steps 2 and 3 to minimize loss of water by evaporation.

Step 3 Reduce the mixer speed to a low level and allow the dispersion to cool to room temperature.

Step 4 Turn the mixer OFF and transfer the oil dispersion to a closed container for storage.

Summary

Ingredients	Total	Nonvolatiles
<i>EASTMAN AQ 55D</i> Polymer (28% solids), parts	100	28
<i>Dow Corning 200</i> Silicone Fluid (350 cS), parts	1400	1400
Water, parts	1356	—
Totals	2856	1428
Nonvolatiles, %		50

Caution: This procedure presents a risk of hot liquid splashes. Operators should wear appropriate protective clothing, including a full face shield and gloves, to cover all exposed skin areas.

II. Dispersing Pellets of *EASTMAN AQ*® Polymer in Water

Example: Prepare 190 liters (50 U.S. gallons) of 30% solids *EASTMAN AQ* polymer dispersion in water.

Step 1 Draw 133 liters (35 U.S. gallons) of soft water (less than 20 ppm total hardness) into a tank or vessel equipped with an agitator. The tank drain should be covered with a screen to prevent pellets from entering the drain pipe. The water should be heated to and maintained in the temperature ranges shown below as pellets of *EASTMAN AQ* polymer are added.

<i>EASTMAN AQ</i> 29S Polymer	75°-85°C (167°-185°F)
<i>EASTMAN AQ</i> 38S Polymer	90°-95°C (194°-203°F)
<i>EASTMAN AQ</i> 55S Polymer	85°-90°C (185°-194°F)

Step 2 Start the agitator. Slowly sift 57 kilograms (125 pounds) of pellets of *EASTMAN AQ* polymer into the water. If the pellets are added too quickly, lumping may occur. Agitate at a rate sufficient to maintain the pellets in suspension.

Step 3 Continue heating in the appropriate temperature range with good agitation until all the pellets are dispersed (usually 20 to 30 minutes). Add water to replace evaporation loss as needed.

Step 4 Cool the dispersion to below 40°C (104°F).

Step 5 Filter before use.

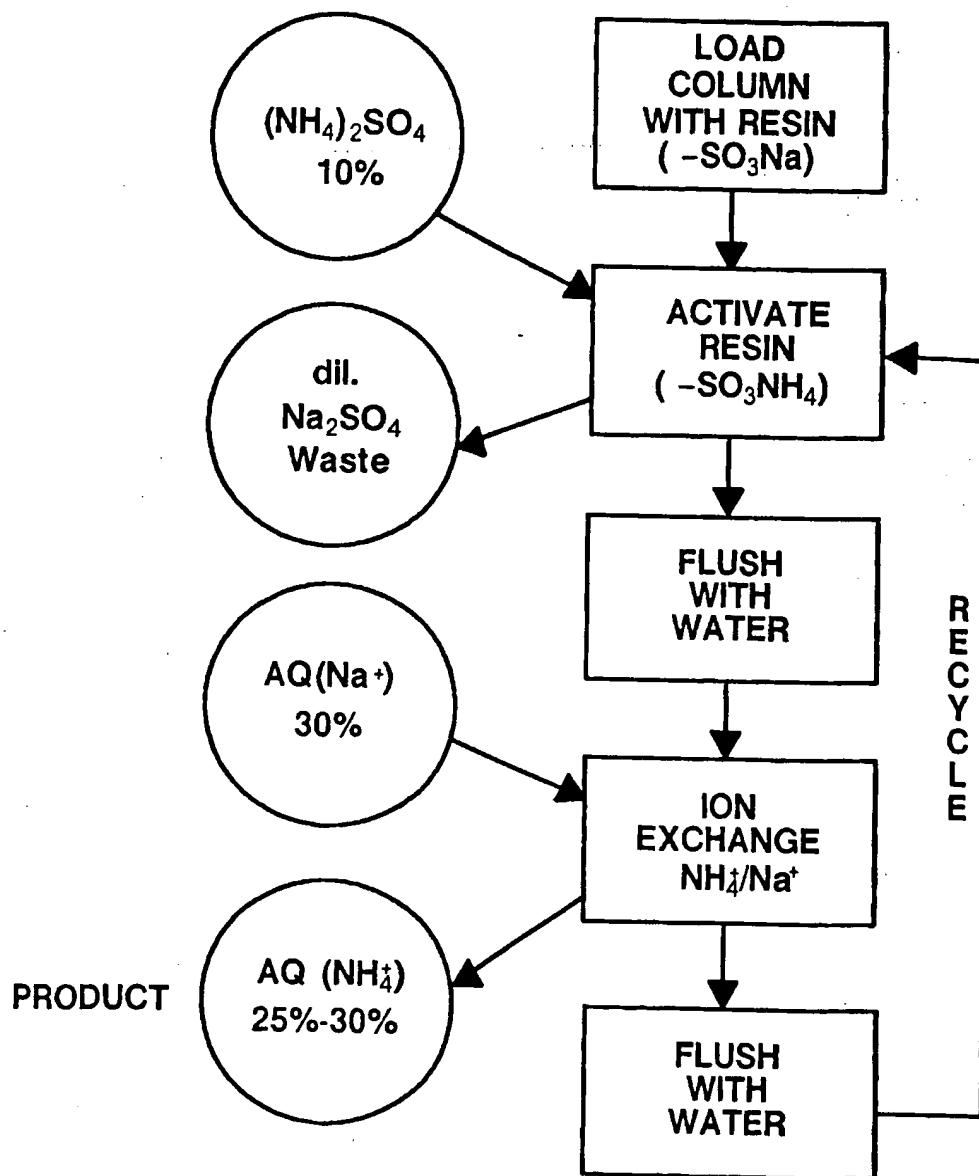
III. Ion Exchange of *EASTMAN AQ*® Polymers

For certain applications such as cross-linkable coatings and PET film priming, it may be desirable to convert the normal sodium salt of *EASTMAN AQ* polymers to an alternate cation form, such as the ammonium salt form. *EASTMAN AQ* polymer salts of several monovalent cations have been produced by the simple ion exchange chromatography procedure outlined below. Attempts to ion exchange *EASTMAN AQ* polymers with divalent and polyvalent cations resulted in plugging of the chromatography column.

Example: Convert *EASTMAN AQ* 29D polymer (Na^+) to *EASTMAN AQ* 29D polymer (NH_4^+)

- Step 1 Load ion exchange column with a strong-acid cation exchange resin such as *Amberlite IR 120 Plus* (Na^+) in water.
- Step 2 Activate the resin bed (i.e., convert resin from $-\text{SO}_3^- \text{Na}^+$ form to $-\text{SO}_3^- \text{NH}_4^+$ form) by passing at least four resin bed volumes of 10% $(\text{NH}_4)_2\text{SO}_4$ solution through the column.
- Step 3 Flush column of excess $(\text{NH}_4)_2\text{SO}_4$ with at least four bed volumes of demineralized water.
- Step 4 Pass *EASTMAN AQ* 29D polymer (Na^+), as supplied, through the activated column to conduct ion exchange. Discard the initial effluent (mostly water) and collect the *EASTMAN AQ* 29D polymer (NH_4^+).
- Step 5 Monitor the Na^+ ion concentration of the effluent with a Na^+ ion-selective electrode and millivolt meter. When the Na^+ concentration in the effluent begins to increase rapidly, the Na^+ breakthrough point has been reached. Stop the column flow. Typically, *EASTMAN AQ* 29D polymer (NH_4^+) generated in this manner will be 30% solids ($\pm 1\%$) and contain less than 20 ppm Na^+ .
- Step 6 Flush the column with at least four bed volumes of demineralized water.
- Step 7 To continue ion exchange with the same resin bed, repeat Steps 2-5.

Ion Exchange Process Diagram



RAW MATERIAL SUPPLIERS

PRODUCT

SUPPLIER

EASTMAN AQ® Polymers
EASTOTAC™ Hydrocarbon Resins
EKTAPRO® EEP Solvent
EKTASOLVE® EB Acetate
EKTASOLVE Solvents
KODAR® PETG Film
TEXANOL® Ester Alcohol

Eastman Chemical Products, Inc.
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Acrysol ICS-1 Acrylic Thickener
Airflex Polymers
Amberlite IR 120 Plus Resin
Aramis Fragrance No. K-1167
Barytes No. 22 Barium Sulfate
Cab-O-Sil M5 Colloidal Silica
Cymel 303 Resin
Dow Corning 200 Silicone Fluid
Duracet Polymers
Flexbond Polymers
Flexcryl Polymers
Hydrated Alumina RH-730
Igepal CO 977 Nonionic Stabilizer
Kadox 515 Zinc Oxide
Kelzan S Xanthan Gum
Lexan Polycarbonate
Lucite Acrylic Sheet
Nacrylic X4280 Acrylic Polymer
Neocryl A-5144 Acrylic Polymer
Neorez R-9431 Urethane Polymer
Resyn 25-1025 PVAc Polymer
Saran Wrap
Shellwax 100 Parafin Wax
Syloid 83 Silica
Ti-Pure R-900 Titanium Dioxide
Vinac XX-230 PVAc Polymer
Vinol 540 Polyvinyl Alcohol
Whiting No. 9 Calcium Carbonate
Witcobond W-260 Urethane Polymer

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05 MAR 1997

Eastman AQ 55S
and
Eastman AQ 38S

POLYESTER POLYMERS FOR COSMETIC APPLICATIONS

EASTMAN

Introduction

Over the years, there has been growing concern regarding the environment and products that damage the stratospheric ozone layer and contribute to ground-level smog. Ozone-depleting substances include chlorofluorocarbons (CFCs) and other volatile organic compounds (VOCs). In 1988, California implemented the Clean Air Act in an effort to protect stratospheric ozone. Effective January 1, 1993, the California Air Resource Board (CARB) began restricting the VOC content of personal care products such as hair spray to a maximum level of 80% for both pump and aerosol products. By January 1, 1998, California will limit all hair spray products to a maximum of 55% VOC content. Table 1 shows CARB regulations for VOC limits and effective dates for specific personal care products. It is expected that there will be an increase in the use of water and other non-VOCs in the production of personal care products because of the need to reduce flammability and VOC content.

Table 1
CARB Regulations for Cosmetic Products

Product	Maximum % VOC	Effective Date
Hair Spray	80	January 1, 1993
	55	January 1, 1998
Hair Mousse	16	January 1, 1994
Styling Gel	6	January 1, 1994
Shaving Cream	5	January 1, 1994

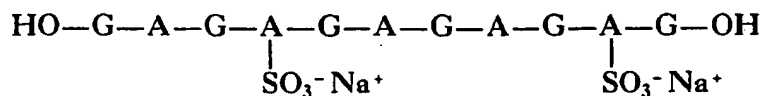
EASTMAN AQ POLYMERS

In response to industry needs, Eastman has introduced *Eastman AQ* polymers, which are novel in that they are:

1. Directly dispersible into water without the aid of surfactants;
2. Thermoplastic film-formers; and
3. Substantive to skin and hair while providing water resistance and low tack.

Eastman AQ polymers are linear polyesters consisting of two types of alternating monomers: aromatic dicarboxylic acids (A) and aliphatic or cycloaliphatic glycols (G). Some of the aromatic dicarboxylic acid groups have sodiosulfo substituents, lending the polymers a slightly anionic character which allows them to be dispersible in water. Figure 1 illustrates the basic structure of the polymer. On average, there are five to eight ionic sodiosulfo substituents per molecule, although only two are depicted in Figure 1.

Figure 1
Basic Structure of *Eastman AQ* Polymer

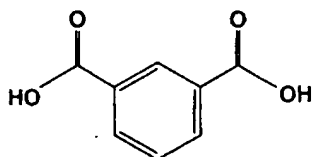


A = an aromatic dicarboxylic acid
G = an aliphatic or cycloaliphatic glycol
—OH = hydroxyl end groups

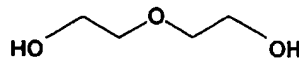
Synthesis of these polymers is achieved via melt-phase condensation polymerization. The molten product is extruded into small, free-flowing pellets. The acids used are typically isophthalic acid (IPA) and 5-sodiosulfoisophthalic acid (SSIPA); the glycols are typically diethylene glycol (DEG) and 1,4-CHDM (cyclohexanedimethanol). These monomers are depicted in Figure 2.

The SSIPA substituents are the primary reason the polymer may be dispersed in water; a secondary contributing factor is the presence of the diethylene glycol.

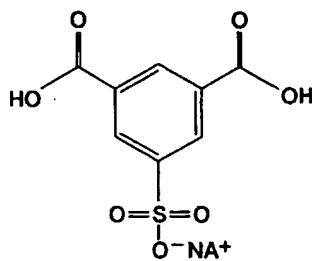
Figure 2
Water-Dispersible Polymers
Chemical Composition



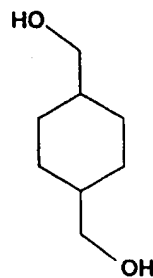
IPA



DEG



SSIPA



CHDM

TYPICAL PROPERTIES

Table 2
Typical Properties^a

	Eastman AQ 55S	Eastman AQ 38S
Percent Solids	100	100
Physical Form	Pellet	Pellet
T _g , °C	55	38
Color	Clear, light amber	Clear, light amber
Inherent Viscosity	0.3–0.4	0.3–0.4
Approximate Molecular wt, M _n ^b		
Polystyrene Standard	2,300	3,000
PET Standard	14,000	14,000

^a Properties reported here are typical of average lots. Eastman makes no representation that the material in any particular shipment will conform exactly to the values given.

^b M_n determinations were made by GPC (gel permeation chromatography) using either polyester or polystyrene standards. Note that M_n determinations by GPC are highly dependent on the type of standard used.

As shown in Figure 1, the end groups are hydroxy groups. However, the hydroxyl numbers are fairly low (usually <10). Carboxyl (—COOH) content is low but detectable; acid numbers are usually <2.

The CTFA designation for *Eastman AQ* polymer is:

diglycol/CHDM/isophthalates/SIP copolymer

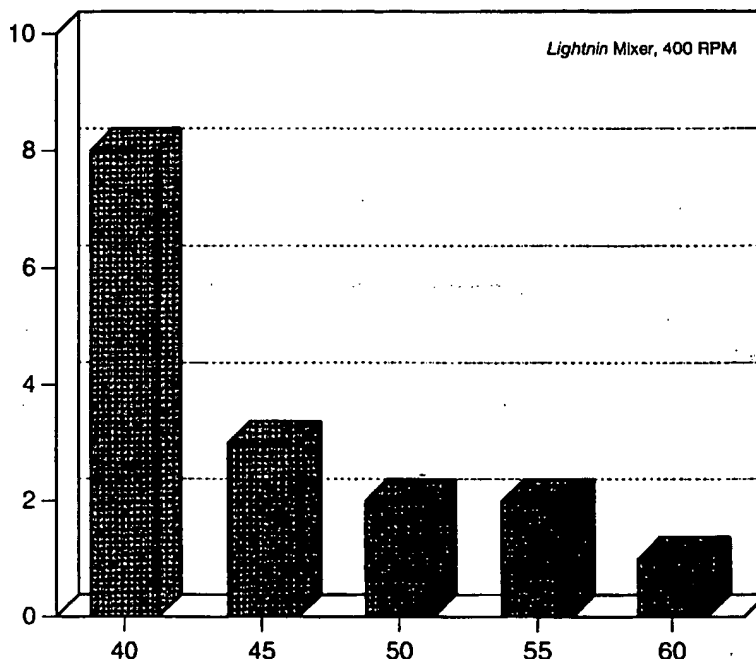
AQUEOUS DISPERSION OF *EASTMAN AQ* POLYMERS

Eastman AQ polymer pellets, as mentioned previously, are directly dispersible (not soluble) in water without the use of surfactants or solvents. A dispersion is defined for the purpose of this publication as being a hydrophobe which is distributed evenly through an aqueous medium. It is also critical that no solid residue be filtered out of dispersion. Dispersions of up to about 30% solids may be easily prepared using deionized water via the following procedure:

1. Heat deionized water (preferably to 60°–80°C).
2. Slowly add pellets to heated water, stirring until uniformly dispersed.
3. Filter dispersion and optionally add biocide.

Deionized water must be used because hard water ions will retard the dispersion rate of the polymer in water and, if present in sufficient concentration, will produce opaque and even unstable dispersions. High concentrations of electrolytes should be avoided; even monovalent electrolytes such as sodium chloride can cause coagulation or gelling of dispersions. The dispersion rate of the pellets is dependent on water temperature, as shown in Figure 3.

Figure 3
Dispersion Rate
Eastman AQ Polymer
(28% Solids)

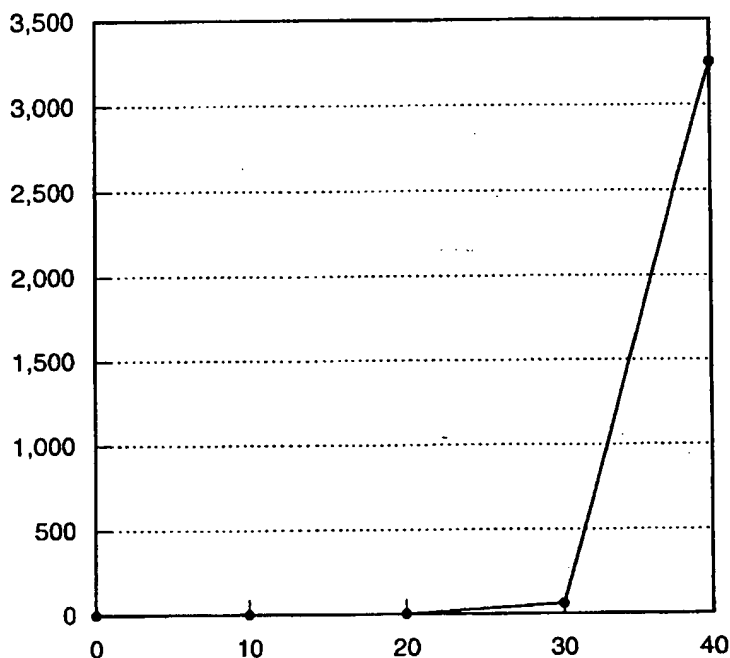


It is possible to produce a dispersion that has the desired % polymer solids or to work from a concentrate diluted with deionized water as necessary to achieve the desired % solids. Dispersions of the polymer are anionic, with a pH of 5–6 at 30% solids. Microscopically, dispersions of these polymers appear as small, discrete spherical particles, with an average diameter of 20 nm, each containing approximately 100 polymer molecules. The polymers in dispersion are most stable at a pH of 5–7. Below pH 5, acid-catalyzed hydrolysis may occur, leading to molecular weight breakdown at high temperatures. Above pH 7, saponification may take place, resulting in the precipitation of nondispersible residue at high temperatures. However, at a pH of 5–7, the molecular weight of the polymer in dispersion remains essentially unchanged even after a year at room temperature.

DISPERSION CHARACTERISTICS

When coated onto a surface, *Eastman AQ* polymer dispersions dry rapidly to form thin, cohesive films having good adhesion without need for coalescing aids or solvents. The rapid dry time is a result of the viscosity versus % solids relationship. The viscosity curve of these dispersions illustrates one of the unique properties of the polymers; i.e., since they are dispersible rather than soluble in water, the viscosity of dispersions remains extremely low up to approximately 30% solids, after which it increases dramatically (see Figure 4).

Figure 4
Viscosity of Dispersion
Based on *Eastman AQ 55S* Polymer



Data depicted in Figure 4 shows that at solids levels of approximately 20% and lower, dispersions have viscosities comparable to water (less than 10 cP), which result in ease of preparation and formulation. However, after approximately 50% of the water is evaporated from the dispersion, it begins to behave as a solid. This is particularly advantageous in water-based systems for which rapid apparent dry time is desirable. Conversely, a dried film of the polymer must absorb a significant amount of moisture from the environment before its properties are adversely affected. Thus, films from *Eastman AQ* polymers exhibit excellent high-humidity resistance.

APPLICATIONS FOR DISPERSIONS OF *EASTMAN AQ* POLYMER

Dispersions of the polymer in water are well suited for a variety of cosmetic applications because of their film-forming, adhesion, and detackifying properties. They impart a good feel to the skin, particularly where a nonoily, non-greasy feel is desired. They are also excellent as hair fixatives. In each of these applications, the exceptional dry times as well as moisture resistance

of the resulting films are advantageous. When formulating products using dispersions, several additives may be incorporated to achieve various properties. Film flexibility, for instance, is dependent on T_g , moisture content, and plasticizer level. Plasticizers such as glycerin or propylene glycol may be used to increase the softness, tackiness, flexibility, and water sensitivity of formulations. The addition of water-soluble polymers may be used to increase flexibility and to aid in shampoo removal. Also, the viscosity of dispersions based on *Eastman AQ* polymers is adjustable using conventional thickeners. However, dispersions are not compatible with cationics, strong acids, or alkalis.

These polymers also have the capacity for dispersing many hydrophobes in water. These hydrophobic materials include waxes, plasticizers, pigments, water-insoluble dyes, sunscreens, and silicone oils. Dispersions of hydrophobes may be achieved using 80 parts oil or 10 to 15 parts wax dispersed by one part polymer at 50% total solids. Hydrophobes that are liquids at room temperature, such as silicone oils, can be dispersed directly into a dispersion of *AQ* polymer with high-shear mixing (i.e., *Kady* mill or *Cowles* blade). For low-melting solids, dispersions are readily obtained by pouring molten wax into a dispersion of the polymer heated to approximately 80°C. At upper loading levels of hydrophobes, these polymers are carriers for the hydrophobe into the aqueous system, resulting in a dispersion without using traditional surfactants. At lower loading levels, the result is a water-borne product somewhat modified by the hydrophobe (i.e., the addition of a fragrance).

SUMMARY

Eastman AQ polymers are easily dispersible directly into water. This makes them especially useful in applications requiring reduced VOC content. The properties of these water-dispersible resins make them extremely useful when formulating hair care products such as hair spray, mousse, setting lotion, and volumizer. In addition, *Eastman AQ* polymers are useful in moisturizing cream, skin cream, sunscreen, mascara, and liquid makeup. The unique properties of these polymers make them useful in a wide variety of cosmetic applications.

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EASTMAN

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